The Effects of Post-Annealing on the Microstructure and Magnetic Properties of Percolated Perpendicular Media

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In this paper the effects of post-deposition annealing on the microstructure and magnetic properties of Co-Pt-SiO_2 percolated perpendicular media (PPM) have been examined. Co-Pt-SiO_2 perpendicular thin films were grown on a Ru underlayer by an alternating sputtering method at room temperature. The as-deposited microstructure is similar to that of Co-alloy/oxide granular media. Desired PPM microstructure was obtained upon post-deposition annealing. The as-deposited films have a low coercivity, whereas high coercivity was found after they were annealed. Transmission electron microscopy investigations indicated that the microstructure of the media did not change much after annealing at temperatures of 600 °C or lower. The desired microstructure and thus good magnetic properties occured after about 5 min at 650 °C. When the annealing temperature is greater than 650 °C and/or the annealing time is longer than 10 min, the SiO_2 pinning sites become larger and less dense.

***Index Terms*—Microstructure, percolated media, perpendicular magnetic media, post-annealing.**

I. INTRODUCTION

THE present granular perpendicular media (GPM) consist of small *hcp* Co alloy magnetic grains that are isolated by an amorphous oxide such as silicon oxide or titanium oxide. The transition jitter noise is dominated by the grain size. Increasing the areal recording density while maintaining sufficient medium signal-to-noise ratio requires a reduction of grain size. However, the size of the magnetic grains is approaching the superparamagnetic limit. As a consequence, the GPM is estimated to have an areal density limitation of less than 500 Gbits/in^2 for today's hard disk drive systems [1].

To be able to keep increasing the areal recording density, a novel thin film microstructure was recently proposed for perpendicular media [2]. This novel medium, which we have denoted as percolated perpendicular media (PPM), consists of closely packed magnetic grains with densely, evenly distributed nonmagnetic entities. The magnetic grains are percolated and hence magnetically coupled. The nonmagnetic entities act as pinning sites for domain walls. The distance between them essentially determines the magnitude of the transition noise. An important property of PPM is that the medium enables a much lower transition noise since the separation between the adjacent pinning sites can be smaller than the grain size in today's conventional perpendicular media, while the ferromagnetic exchange coupling in the medium enables sufficient thermal magnetic stability because the medium is continuous and hence all the grains are exchange coupled. A comprehensive micromagnetic simulation study has shown that PPM can offer much improved recording properties over the present GPM [3]. A first attempt to fabricate and measure the magnetic properties of PPM has been reported on the Co-Pt-SiO_2 two phase thin films [4]. Preliminary results showed that PPM is a very promising medium. However, more experimental work is needed to optimize the microstructure and thus to improve the magnet properties of the medium.

In this research, we investigate the effects of post-deposition annealing on the microstructure and magnetic properties of the Co-Pt-SiO_2 PPM.

II. EXPERIMENT

Co-Pt-SiO_2 films and Ru/Ta underlayers were deposited on Si substrates by RF magnetron sputtering. The Co-Pt-SiO_2 films were fabricated by alternate sputtering from Co-16%Pt and SiO_2 targets, as described elsewhere [4]. The volume fraction of oxide can be controlled by the relative sputtering times. After deposition, samples were annealed by rapid thermal annealing (RTA) at temperatures in the range of 550 °C–700 °C for 1–30 min. X-ray diffraction (XRD) and transmission electron microscopy (TEM) were used to study the microstructure of the films. An alternating gradient field magnetometer (AGFM) was used to measure the magnetic properties.

III. RESULTS AND DISCUSSION

The as-deposited Co-Pt-SiO_2 films have a very low coercivity. After annealing, the coercivity increased for all samples except those with a SiO_2 volume fraction greater than 27%. In order to understand this behavior, TEM was used to investigate the microstructure of the films. A typical plan-view image from a Co-Pt-7.7%SiO_2 film is shown in Fig. 1(a). It has a similar microstructure as the current perpendicular media, i.e., it consists of small Co-Pt grains surrounded by amorphous oxide. The microstructure of this specific oxide fraction media shows a grain size of about 14.2 nm and an oxide thickness of about 1.5 nm. Current media have grains of about 6-10 nm and oxide thickness less than about 1 nm. The magnetic film with this microstructure has a low coercivity, likely due to incomplete segregation of SiO_2 to the grain boundaries, which could result in insufficient intergranular exchange decoupling [5]. After annealing at 650 °C for 5 min, the coercivity increases dramatically to a value of about 3.5 kOe. The microstructure of the annealed

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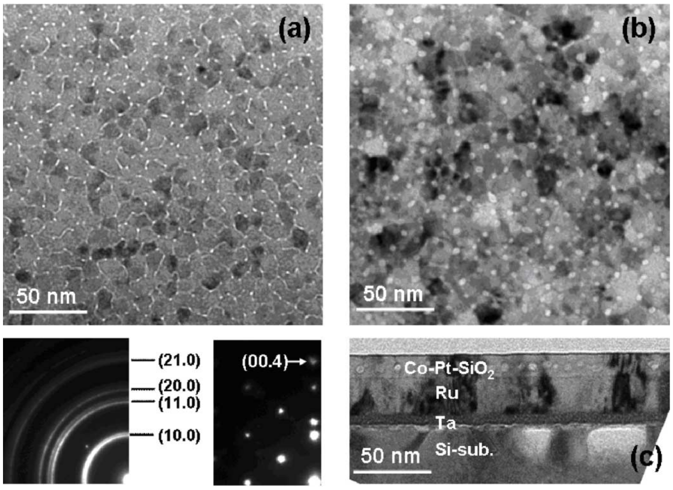

Fig. 1. TEM images of a Co-Pt-7.7% SiO_2 thin film. (a) Plan-view image of the as-deposited film. (b) Plan-view image of the annealed film. (c) Cross-section image of the annealed film. The electron diffraction patterns from both plan-view and cross-section confirm the (00.2) film texture structure.

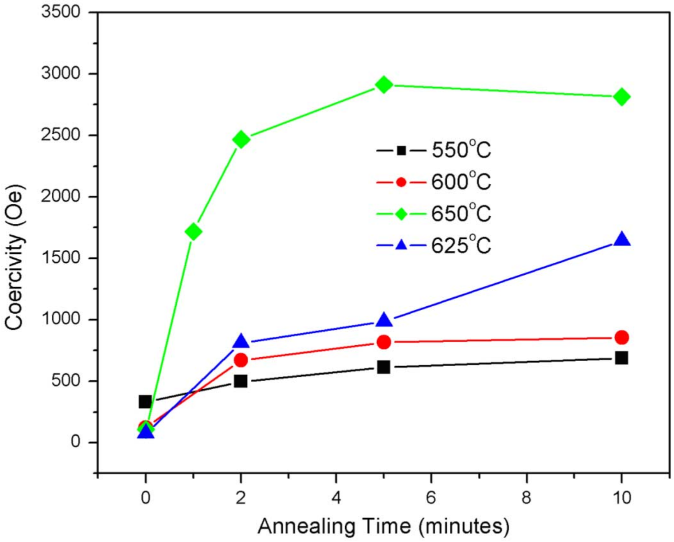

Fig. 2. Variations of the coercivity with annealing temperature and annealing time for a Co-Pt-9% SiO_2 thin film.

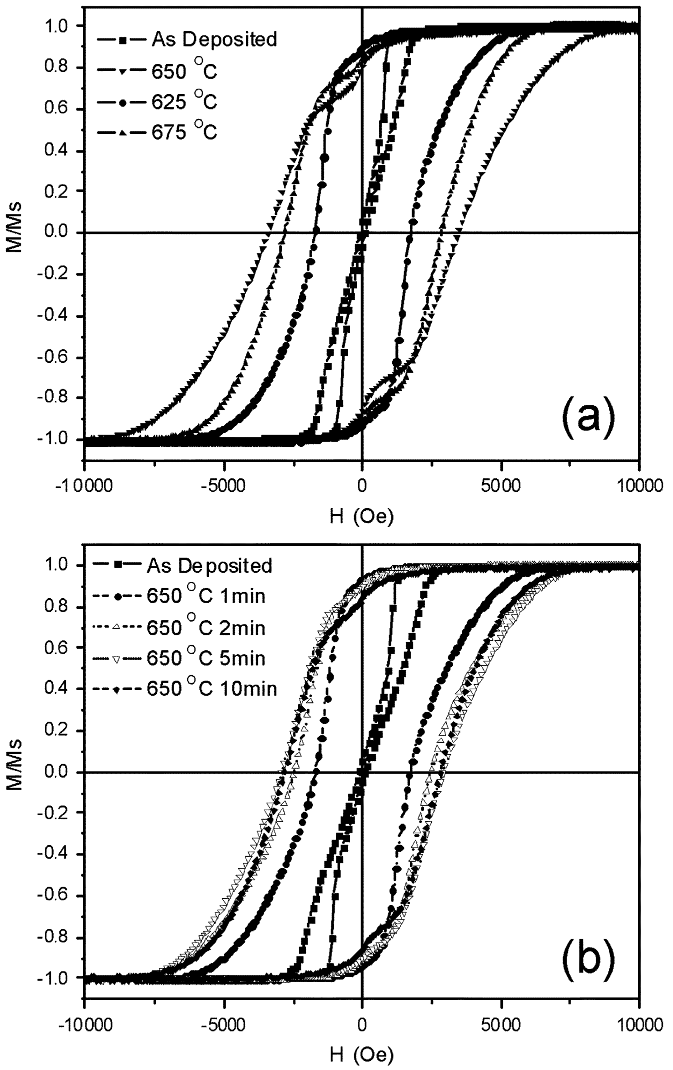

Fig. 3. (a) M-H loops of the Co-Pt-7.7% SiO_2 thin films of the as deposited and after annealing at various temperatures for 5 min. (b) M-H loops of the Co-Pt-9% SiO_2 thin films of the as deposited and after annealing at 650 °C for 1–10 min. The thickness of the media films is 15 nm. The applied field is perpendicular to the film plane.

sample is shown in Fig. 1(b) for the plan-view and Fig. 1(c) for the cross-section, respectively. The magnetic grains are magnetically interconnected, while the oxide forms spherical particles in the middle of the film, which have a diameter of about 4–5 nm and a spacing of about 10.3 nm. The *hcp* Co-Pt grains remain highly textured crystallographically, with their hexagonal *c*-axes perpendicular to the film. The Co-Pt grain size of the annealed films is slightly larger than that of the as-deposited ones. The oxide phase pins the magnetic domain walls, hindering their motion and hence producing the increased coercivity.

Fig. 2 shows the effects of annealing temperature and time. Heating the sample at lower temperature than 600 °C even for a long time does not change the coercivity very much. The coercivity increases dramatically only at higher temperatures for a short time. Hysteresis loops of a Co-Pt-7.7%SiO_2 film shown in Fig. 3(a) indicates that the best annealing temperature is at around 650 °C. Fig. 3(b) shows another Co-Pt film with a 9% SiO_2, which annealed at 650 °C for various time. It can be seen that 1-min annealing has caused a significant increase in the coercivity. Five-min annealing gives the highest coercivity. Longer time annealing does not improve the coercivity further. Therefore, the suitable annealing time is at 650 °C around 5 min.

To understand the effects of the annealing time, TEM microstructural investigations were performed on this film. Fig. 4 shows the microstructures of the as-deposited thin film and that annealed at 650 °C for 1, 5, and 10 min, respectively. It can be seen that 1-min annealing causes most amorphous oxide in the granular microstructure [Fig. 4(a)] to agglomerate [Fig. 4(b)], but not completely until after 5 min [Fig. 4(c)]. Upon further annealing, the isolated spheres grew into a larger size and became less dense [Fig. 4(d)].

XRD patterns displaying the (00.2) peaks of the hcp Ru underlayer and Co-Pt thin film as a function of annealing times are shown in Fig. 5. The films show a highly textured microstructure which remained after the annealing. The *c* lattice parameter

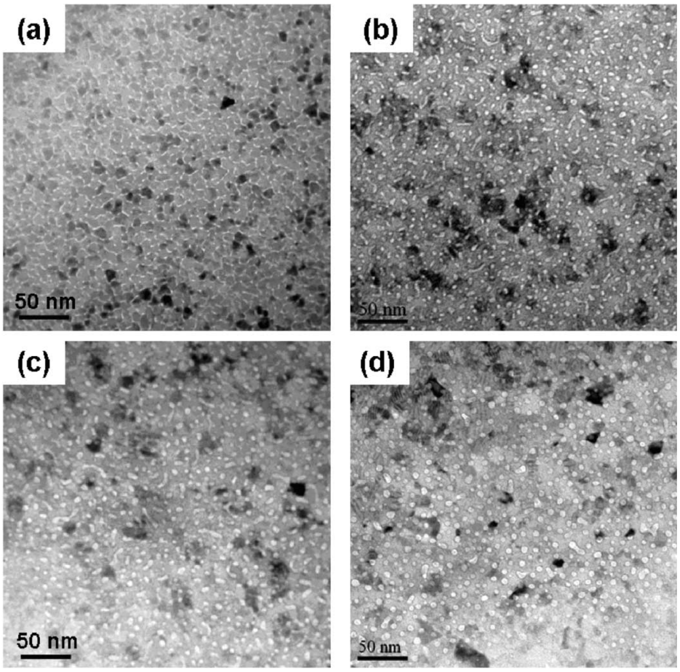

Fig. 4. Plan-view TEM micrographs of Co-Pt-9% SiO_2 thin films. (a) As deposited, and after annealing at 650 °C for (b) 1 min, (c) 5 min, and (d) 10 min.

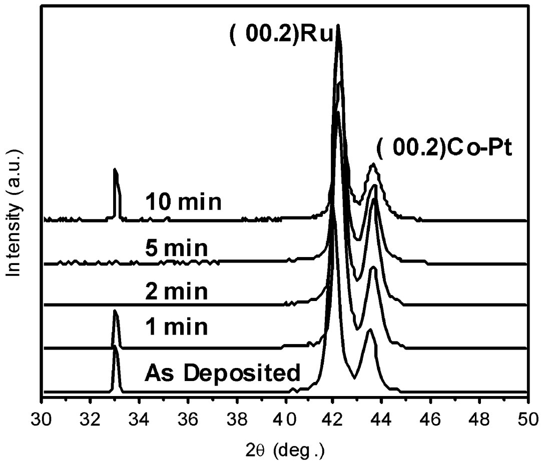

Fig. 5. Conventional $2\theta/\theta$ XRD spectra for a 15 nm CoPt-9% SiO_2 media layer on a 20 nm Ru underlayer and after annealing at 650 °C for various time.

of the films decreased and approached their bulk values after the annealing.

Previous work showed that the granular microstructure represented in the as-deposited films has a high oxide/metal interface energy which can be reduced by changing the microstructure to the percolated microstructure shown in Fig. 1(b) [4]. It was assumed that the oxide first break into separated cylinders. If the samples are heated for extended times, the cylinders would break up by the Rayleigh instability [6], [7] into spherical regions, giving rise to more, but weaker pinning sites. Therefore, the optimum microstructure for PPM is only at an intermediate stage, so care must be taken to stop the evolution of the oxide microstructure at the point where cylinders occur. Thus, the annealing temperature and time must be carefully controlled to obtain the optimum microstructure. As indicated above, the best annealing parameters were 650 °C for 5 min.

Another concern is the density of the pinning sites, i.e., the separation between adjacent pinning sites, which is controlled by the Co-Pt grain size in the fabrication process described in this paper. Therefore, further work is needed to obtain smaller Co-Pt grains. Moreover, since our current fabrication process involves high temperature annealing, which is unfavorable for the industry applications, it is important to be able to produce the PPM microstructure without post-annealing.

IV. CONCLUSION

Desired PPM microstructure can be obtained upon post-deposition annealing from the as-deposited films. However, care should be taken for the annealing process. For our films, the best annealing parameters were found to be 650 °C for 5 min. TEM showed the evidence that the high density oxide pinning sites exist among the Co-Pt thin films. When the annealing temperature is higher than 650 °C or the annealing time is longer than 10 min, the SiO_2 pinning sites became bigger but less dense. More work is still needed to optimize the PPM microstructure.

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REFERENCES

[1] J.-G. Zhu, Data Storage Systems Center Annual Review, 2005.
[2] J.-G. Zhu and Y. Tang, "A medium microstructure for high area density perpendicular recording," *J. Appl. Phys.*, vol. 99, p. 08Q903, 2006.
[3] Y. Tang and J.-G. Zhu, "Micromagnetics of percolated perpendicular media for 1 Tbits/in^2 and beyond," *IEEE Trans. Magn.*, vol. 42, no. 10, pp. 2360–2362, Oct. 2006.
[4] D. E. Laughlin, Y. Peng, Y. L. Qin, M. Lin, and J.-G. Zhu, "Fabrication, microstructure, magnetic and recording properties of percolated perpendicular media," *IEEE Trans. Magn.*, vol. 43, no. 2, pp. 693–697, Feb. 2007.
[5] J.-G. Zhu and H. N. Bertram, "Magnetization reversal in CoCr perpendicular thin films," *J. Appl. Phys.*, vol. 66, pp. 1291–1307, 1989.
[6] L. Rayleigh, in *Proc. London Math. Soc.*, 1879, vol. 10, p. 4.
[7] F. A. Nichols and W. W. Mullins, *Trans. Metall. Soc., AIME*, vol. 233, p. 1840, 1965.

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